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Condensed-Phase Processes During Solid Propellant Combustion. IV. Chemical and Microscopic Examination of Laser-Irradiated Propellant Samples

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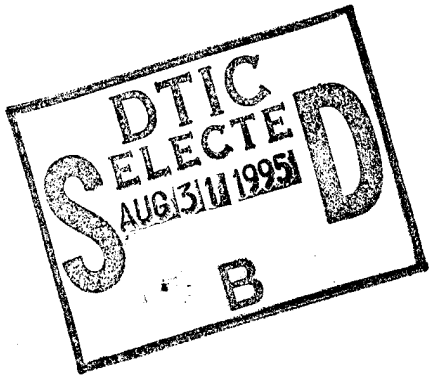

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The surfaces of laser-irradiated gun propellants have been examined by scanning electron microscopy (SEM) and Fourier Transform Infrared (FTIR) microscopy. The results suggest that the laser-irradiated surfaces exhibit chemical and physical changes similar to those exhibited by the burned surfaces of propellant samples, the combustion of which have been quenched short of completion. Some possible implications of this for combustion and for the modelling of laser ignition are discussed.

14. SUBJECT TERMS

propellants, gun propellants, XM39, M9, M10, M30, JA2, laser ignition, laser pyrolysis, combustion, ignition, pyrolysis, burning surfaces

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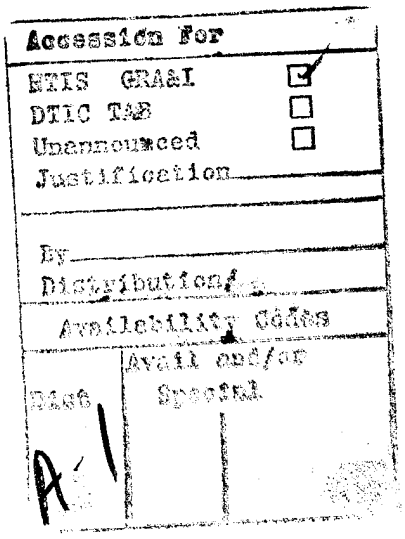
PREFACE

This report is the fourth in a series of reports examining the chemical and microstructural nature of several burned propellant samples. While this investigation was in progress, the U.S. Army Ballistic Research Laboratory (BRL) was deactivated and became part of the U.S. Army Research Laboratory (ARL).



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ACKNOWLEDGMENT

The authors are grateful to Robert J. Lieb for helpful discussions and for the use of some of his equipment, including a drop-hammer apparatus for splitting propellant grains. The work described here could not have been accomplished without equipment provided by the Productivity Enhancement Capital Investment Program.

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1. INTRODUCTION

This is a progress report on work aimed at understanding the nature and importance of condensed phase reactions in the combustion of nitramine and other solid gun propellants. Information on the nature and importance of condensed phase reactions is needed as input for modelling studies. This information could also be very important in understanding the relationship of chemical structure and of physical properties such as melting points, phase transition temperatures, etc., to explosive and propellant behavior.

Our previous work on burned propellants has been described in three reports (Schroeder et al. 1990, 1992, and 1994). In that work, burning propellant samples were quenched, and the burned surfaces were examined both microscopically and by chemical analysis (Schroeder et al. 1990, 1992). Studies (Schroeder et al. 1990, 1992) were carried out on a series of propellants and compositions including XM39, M30, JA2, RDX, and the HMX/binder composition HMX2. Preliminary results (Schroeder et al. 1990) on XM39 were consistent with the idea that at least at low pressures there is a liquid layer approximately 100–300 μm thick in combustion of nitramine propellants; scanning electron microscopic (SEM) examination revealed little if any evidence of degradation below this layer. Gas chromatography mass spectroscopy (GCMS) analysis (Schroeder et al. 1990, 1992) suggests that the stabilizer is considerably depleted relative to the plasticizer in the surface layers, presumably by reaction with nitrogen oxides formed by decomposition of RDX and NC. High-performance liquid chromatography (HPLC) results suggest that there is a significant increase in concentration of the mechanistically significant nitrosoamines MRDX and DRDX over the very small amounts possibly present as impurities in RDX. The structures of these nitrosoamines are similar to that of RDX, with respectively one or two nitro groups replaced by nitroso groups. These results are consistent with the idea that RDX decomposition under propellant combustion conditions involves primarily (a) vaporization followed by gas-phase decomposition and/or (b) decomposition in the liquid phase to products that are almost entirely gaseous. Photoacoustic Fourier transform infrared (FTIR-PAS) examination (Schroeder et al. 1990, 1992) of the burned surfaces also provided evidence for condensed-phase reaction in the compositions studied. Results of SEM examination of the burned surfaces of quenched samples of HMX2, HMX/PU, RDX, M30, and JA2 were also presented (Schroeder et al. 1990, 1992). Some depth-profiling results have also been reported (Schroeder et al. 1994).

In the present report, a somewhat different departure is described. A series of laser-irradiated propellant samples have been obtained in the course of studies (Beyer and Cohen 1992) on laser ignition

of gun propellants. Each of these samples consists of a grain of propellant marked by a cavity several millimeters wide and several millimeters deep where the grain was struck by the laser beam. During laser irradiation, the sample appeared to burn, but combustion ceased on deradiation. The inside surfaces of the cavities are being examined by SEM, optical microscopy, and by chromatographic and spectroscopic methods.

The reasons for studying these samples are as follows: (a) they are pertinent to our work because it seems relevant to our understanding of the combustion process to see whether the effect of exposing a propellant surface to any external heat source, such as a laser, will be similar to the effect of the combustion process as revealed by our previous studies; (b) our access to the strand burner needed for the continuation of our regular work was delayed due to the press of other work; and (c) an examination of these surfaces was of interest to workers at ARL who are engaged in the study of laser ignition.

2. EXPERIMENTAL

Laser irradiation (Beyer and Cohen 1992) of the samples was carried out on a continuous-wave CO_2 (10.6 μm) laser (Synrad model #57) with variable power (nominal 10–100 W) used as the ignition source. Data were obtained for the following propellants: M9 (homogeneous double base), M30 (composite triple base), XM39 (RDX composite), and HMX2 (HMX composite). The samples were small disks approximately 0.19 in wide. The M30 and XM39 disks were cut from 0.25-in-diameter grains (perforated and with graphite surfaces), and the others were cut from 0.25-in-diameter strands. The beam, having about a 0.118-in diameter, was directed along the propellant axis and covered approximately 25% of the sample surface. Pulse lengths were varied from 0.1 to 15 s. Irradiation was carried out in either open air or in a closed 1-ℓ bomb.

The irradiated grains were cooled to dry ice temperatures and split with a knife blade held vertical by mechanical means; when this knife blade was rested against the propellant grain and struck by a hammer, a clean split could be obtained (the knife was mechanically prevented from penetrating more than a small fraction of the grain). One half of the split grain was kept for spectroscopic and chromatographic examination, and the other half was examined by SEM.

The SEM microscope used was a JEOL Model JSM-820 instrument. Samples were sputtered with gold before examination.

FTIR-microscope spectra were obtained with 8-cm^{-1} resolution and 1,024 scans, using a Spectra-Tech IR-Plan infrared microscope, operating in reflectance mode and interfaced to a Mattson Polaris FTIR spectrometer operated through a PC running Mattson FIRST software. Where necessary, the Kramers-Kronig transformation was used to remove spectral distortions. Spectra were obtained on unirradiated propellant surfaces and on the center (bottom) surface of the crater made by the laser beam. The area sampled was a square 400 μm on a side.

3. RESULTS

The samples examined were taken from a series of samples that had been irradiated in connection with studies in progress at ARL (Beyer and Cohen 1992). These samples had been irradiated with a laser of known power for known lengths of time; during irradiation a jet of flame shot out from the sample, but it disappeared on deradiation (i.e., when the laser was turned off), leaving a crater where the sample was struck by the laser beam. A large number of samples were available; however, because of time limitations, only a limited number were chosen. For the preliminary study described here, a series of samples with moderate damage (medium-sized craters) was chosen and examined by SEM and by FTIR microscopy.

Table 1 gives the compositions of the propellants studied, and Table 2 gives the conditions under which laser-irradiation of the various samples took place.

SEM microscope photos are shown in Figures 1–11, and FTIR spectra are shown in Figures 12–16.

Our observations from the study of these samples are summarized in Table 3. FTIR-microscope examination of the M10 and M30 samples has not yet been carried out.

4. DISCUSSION AND CONCLUSIONS

4.1 Examination of Laser-Irradiated Samples: SEM. Figures 1 and 2 respectively show an overall view of a laser-irradiated grain (no. 33, Table 2) of XM39 propellant, and a close-up view of part of the melt layer. The surface is covered with debris that looks as though it might be decomposed binder, and there is a melt layer which contains numerous bubbles and averages several hundred micrometers in

Table 1. Compositions of Propellant Formulations Studied

Propellant	Ingredient	Composition (%)
XM39	RDX	76.0
	Cellulose Acetate Butyrate (CAB)	12.0
	Acetyl Triethyl Citrate (ATEC)	7.6
	Nitrocellulose (NC) (12.6% N)	4.0
	Ethyl Centralite (EC)	0.4
M30	Nitroguanidine (NQ)	47.7
	Nitrocellulose (NC) (12.68% N)	28.0
	Nitroglycerine (NG)	22.5
	Ethyl Centralite (EC)	1.5
	Cryolite	0.3
JA2	Nitrocellulose (NC) (13.04% N)	59.5
	Diethylene Glycol Dinitrate (DEGDN)	14.9
	Nitroglycerine (NG)	24.8
	Ethyl Centralite (EC)	0.7
	Magnesium Oxide	0.05
	Graphite	0.05
M9	Nitrocellulose (NC) (13.29% N)	57.75
	Nitroglycerine (NG)	40.00
	Ethyl Centralite	0.75
	Potassium Nitrate	1.50
M10	Nitrocellulose (NC) (13.16% N)	98.00
	Potassium Sulfate	1.00
	Diphenylamine	1.00

Table 2. Conditions of Laser Irradiation of the Samples Studied

Sample No. and Propellant	Atmosphere	Laser Power (W)	Time (ms)
24 (JA2)	Air	1.7	2,700
33 (XM39)	Air	1.2	1,216
46 (M30)	Air	1.7	1,628
58 (XM39)	Air	41.0	384
90 (M9)	Air	114.0	117
105 (M10)	N_2	34.3	260
106 (M10)	Air	34.3	300
115 (M9)	N_2	1.8	1,082

Table 3. Summary of Observations for Compositions Studied

Propellant	Melt Thickness (μm)	General Conclusions (This Study)
XM39	100–300	Surface Color: Off-white to yellow/orange at bottom, black inside rim. Melt layer contains bubbles and signs of crystallization and is covered in places with debris. FTIR-Microscopy: Shows increase in carbonyl (>C = 0) peak due to binder and its decomposition products.
M30	10–20	Surface Color: Dirty brown, much less black inside rim than in the case of XM39. Melt layer contains bubbles and is covered in places with debris.
M9	10	Surface Color: Same as bulk propellant. Melt layer smooth, contains some bubbles and cracks. FTIR-Microscopy: Irradiation causes appearance of >C = 0 peak.
M10	10	Surface Color: Dirty brown or (one sample) yellow green; some flecks of brown. Melt layer has more bubbles than in case of M9.
JA2	10	Surface Color: Same as bulk propellant (yellow-orange). Melt layer has more bubbles than in case of M9. FTIR-Microscopy: Irradiation causes appearance of >C = 0 peak.

thickness. The bubbles presumably arise from decomposition, especially of RDX, and are probably indicative of condensed-phase decomposition of RDX oxidizer. A closer view of the melt layer (Figure 2) shows the presence of what appear to be crystals, presumably from crystallization of molten RDX on cooling and solidification. The irradiated surface layers here resemble those (Schroeder et al. 1990) of XM39 propellant burned in air at atmospheric pressure and quenched by dropping in water.

Figures 3–5 respectively show an overall view and some closeups of a laser-irradiated grain (no. 46, Table 2) of M30 propellant. A cross section of the burned surface (Figure 4) shows a very thin bubbly melt layer, at most 10–20 μm thick. As in the case of XM39, the bubbles presumably arise from decomposition and are probably indicative of condensed-phase decomposition. The top of the surface appears bubbly (Figure 5) and is partially covered with debris (Figure 3). Thus, as in the case of XM39, the irradiated surface of M30 generally resembles that (Schroeder et al. 1992) of M30 propellant burned in air at atmospheric pressure and quenched by dropping it in water. One difference is the presence of

small amounts of fuzzy material in places on the surface of the laser-irradiated M30 sample; this is possibly nitroguanidine or its decomposition products that have sublimed, then condensed on quenching by deradiation.

Figures 6 and 7 respectively show an overall view of an irradiated grain of M9 propellant and a closeup of the irradiated surface. The "hills" or "steps" inside the crater (Figure 6) are possibly the result of some inhomogeneity in the laser beam. The surface appears generally smooth, although covered in places with bubbles (condensed-phase decomposition), cracks, and flecks of debris. The melt layer appears very thin, about 10 μm at most; this is consistent with the results of depth profiling studies (Schroeder et al. 1994) on M9.

Figures 8 and 9 respectively show an overall view of a laser-irradiated grain of M10 propellant and a closeup of the cross-section of the burned surface. The surface appears covered with bubbles (condensed-phase decomposition), and the melt layer is quite thin, no more than 10 μm or so thick; these observations are consistent with the behavior of the nitrate ester compositions M9 and JA2 in combustion (Schroeder et al. 1992, 1994) and laser-irradiation (present work) conditions.

Figures 10 and 11 respectively show an overall view of a laser-irradiated grain of JA2 propellant and a cross section of the melt layer taken on the same grain. Again, the surface appears covered with bubbles (condensed-phase decomposition), and the melt layer is quite thin (no more than 10 μm or so thick); this is consistent with the behavior of the nitrate ester compositions M9 and M10 under combustion (Schroeder et al. 1992, 1994) and laser-irradiation (present work) conditions.

4.2 Examination of Laser-Irradiated Samples: FTIR Microscopy. Figure 12 shows an FTIR-microscope spectrum of the laser-irradiated surface of a grain of M9 propellant, and Figure 13 shows a comparison among a laser-irradiated grain of M9 Propellant, a burned/extinguished (Schroeder et al. 1994) grain of M9 propellant, and an FTIR-microscope spectrum of virgin M9. The changes in the spectra on laser irradiation and on burning/extinguishment are almost the same, consisting of the appearance of weak absorption in the carbonyl region at approximately 1,730 cm^{-1} and possibly of a slight increase in the C-O-C bands in the 1,000- to 1,200-cm^{-1} region.

Figure 14 shows a similar comparison for JA2 propellant. The spectrum quality of the laser-irradiated sample is not as good as the others, but the appearance of the carbonyl (>C = O) band in the 1,730-cm^{-1}

region can be seen. These changes are believed to be due to loss of NO_2 due to O-N cleavage of the nitrate ester groupings during initial decomposition of the nitrate ester ingredients of these propellants (Table 1); this would result in a decrease in intensity of the NO_2 bands relative to the skeletal C-O-C bands; furthermore, the alkoxy radical left behind might be expected to ultimately lead to an aldehyde or ketone grouping, having (Gordon and Ford 1972; Silverstein, Bassler, and Morrill 1991) characteristic absorption in the region around 1,720 cm^{-1}.

Figure 15 shows FTIR-microscope spectra of virgin XM39 propellant, and Figure 16 shows two different irradiated grains of XM39 propellant. Note that although they differ in similar ways from the spectrum of virgin XM39, the two spectra of irradiated XM39 are not identical to each other; thus it appears that the duration and intensity of the laser pulse have some effect on the extent of the chemical changes observed by FTIR microscopy. Comparison with the surface spectra (Schroeder et al. 1994) of burned/extinguished samples suggests that the changes on irradiation and on burning tend to be similar but that more RDX remains on the surface of the laser-irradiated samples. These changes are possibly due to accumulation of the CAB binder and its decomposition products at the burning surface as the decomposition proceeds, as described in Schroeder et al. (1994). Note, however, that the possible effect of differences in absorption coefficients of the RDX and of the binder for the laser light has not been examined.

4.3 Comparison of Laser-Irradiated and Burned/Extinguished Samples. The SEM studies indicate that with regard to melt layer thickness, presence of bubbles, etc., the results to date are in general agreement with our earlier results (Schroeder et al. 1990, 1992, and 1994) for the surfaces of burned/quenched propellant samples.

In addition, the results of FTIR-microscope analysis of the inside, laser-irradiated surfaces of the cavities suggest that the chemical changes in the irradiated layers are similar in nature to those observed earlier for quenched surfaces of burned/quenched propellant samples.

The results described here seem most consistent with the idea that the principal interaction of the laser beam with the sample is thermal in nature. This is suggested by (a) the presence of the melt layer in all samples, as shown by the presence of bubbles and (in the case of XM39) of crystallization of melted RDX; and (b) the similarity in appearance between the burned/extinguished surfaces (Schroeder et al. 1990, 1992) and the laser-irradiated surfaces described here.

The similarity between laser-irradiated and burned/extinguished propellant surface layers is of interest because it suggests that, in both cases, the changes in the surface layers are caused by the imposition of a heat source from above. In the case of the burned/extinguished samples, the heat is generated by the reaction of gaseous products of decomposition of the propellant ingredients, while in the case of the laser-irradiated samples, the external heat is presumably due partly to the laser beam itself and partly to heat generated by reaction (combustion) between gaseous products of laser-induced decomposition of the propellant ingredients. This makes sense if, as seems reasonable (Ostmark and Grans 1990; Ostrowski et al. 1980) (see also the preceding paragraph), the effect of the laser on these samples was primarily thermal in nature.

Another interesting point from these studies is that they demonstrate the presence of a melt layer in these samples. Since models of laser ignition have had only partial success (Beyer and Cohen 1992) in predicting times to emission, times to flamespreading and times to pressure buildup, this suggests that it might be helpful to modify existing laser ignition models to take into account the existence of the liquid layer.

5. RECOMMENDATIONS FOR FUTURE RESEARCH

Further laser-irradiated samples should be examined in order to evaluate the effect on the results of such factors as atmosphere, laser-pulse power, and duration.

Runs should be carried out in a strand burner with depressurization-quenching, and the results should be compared with those from the quenching methods used to date.

Optical microscope examination of the burned surfaces should be carried out, particularly in view of the possibility that studies of color changes and variations may yield information on the occurrence (or lack thereof) of chemical changes in the solid below the liquid layer.

Additional methods for depth-profiling of the burned layers are being explored; these include microabrasive blasting, microtoming, solvent-dipping, and improved scraping procedures. Depth-profiling should be applied to the nitrosoamine-formation and stabilizer-depletion results reported previously (Schroeder et al. 1990, 1992). Additional binders, oxidizers, and formulations should be studied.

Isotope-scrambling studies on burning (rather than merely decomposing) samples are needed. These would involve use of mixtures of unlabelled RDX or HMX with RDX or HMX labelled with nitrogen-15 in all nitrogens, both in the ring and in the nitro groups. Use of these mixtures would lead to scrambled nitrosoamines if the recombination mechanism were operating, but to unscrambled nitrosamines if an oxygen-abstraction mechanism were operating. Partial scrambling might mean that both mechanisms were operating to some degree. Note, however, that while formation of fully scrambled nitrosoamines would provide no evidence for oxygen abstraction, it would not necessarily rule it out, since the scrambling could have taken place by further N-N cleavage equilibria before or after formation of the nitrosoamines.

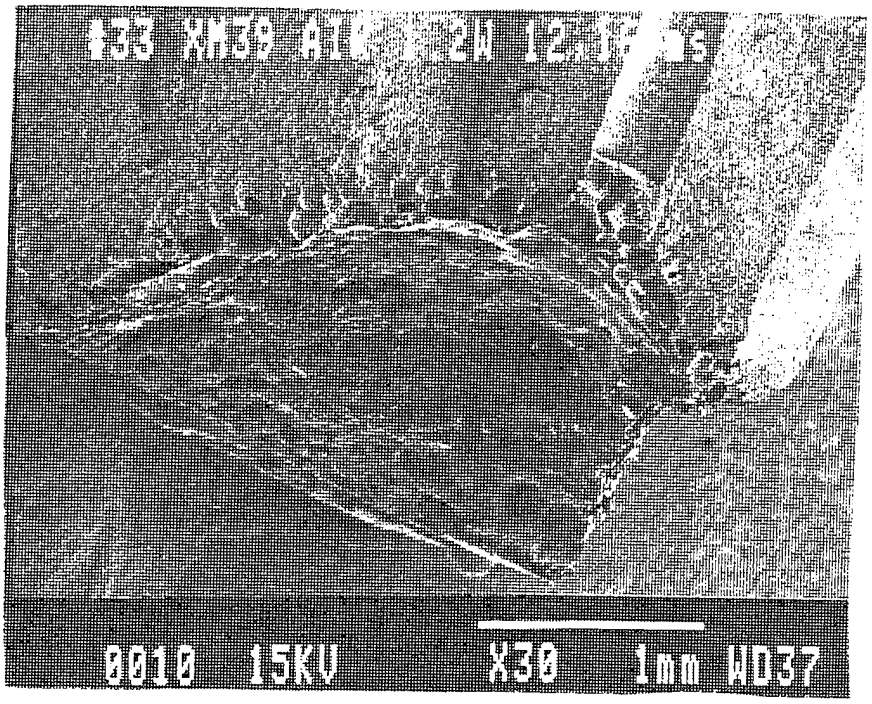

Figure 1. Overall view of portion of laser-irradiated grain of XM39 propellant.

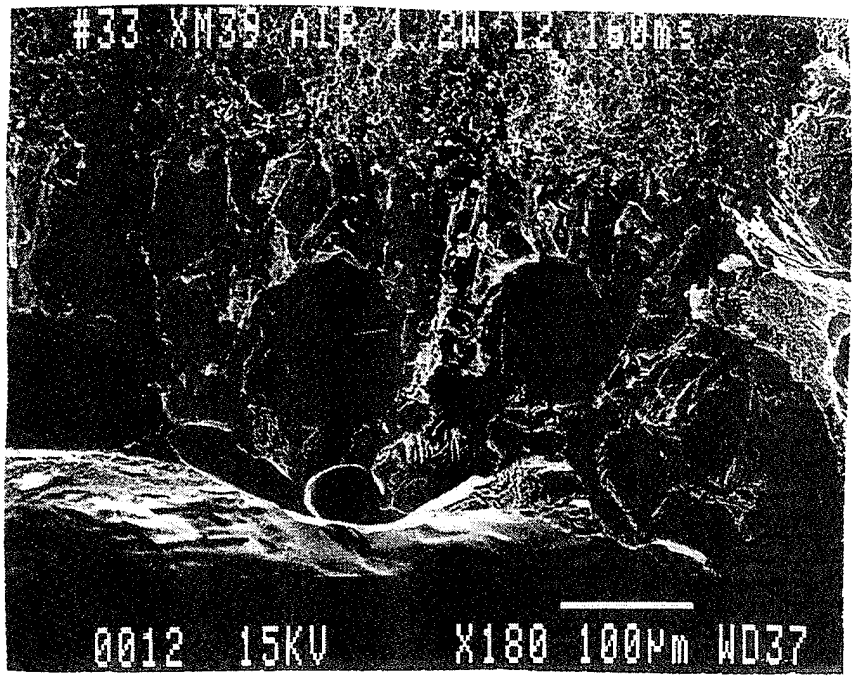

Figure 2. Cross section of melt layer of laser-irradiated grain of XM39 propellant.

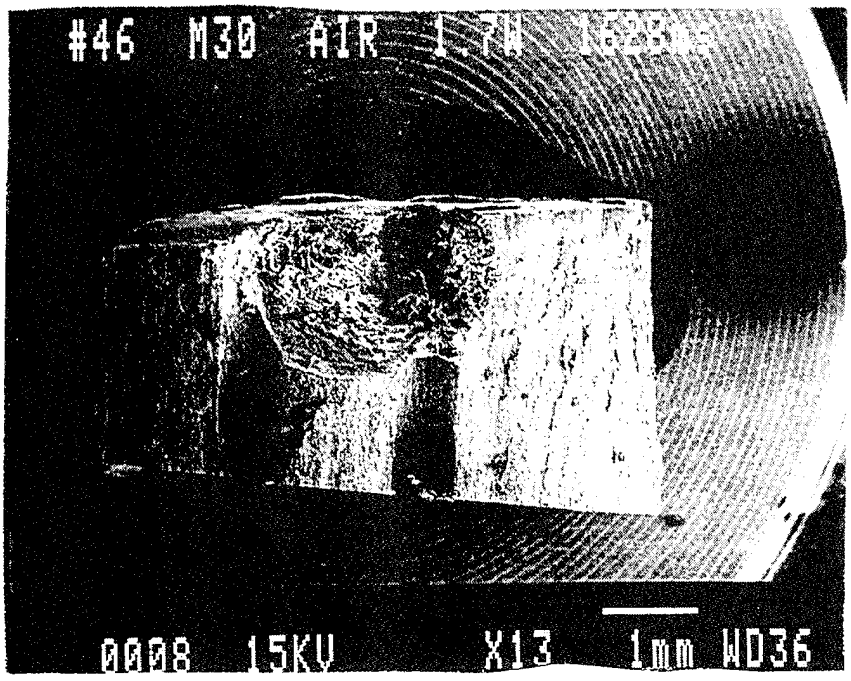

Figure 3. Overall view of portion of laser-irradiated grain of M30 propellant.

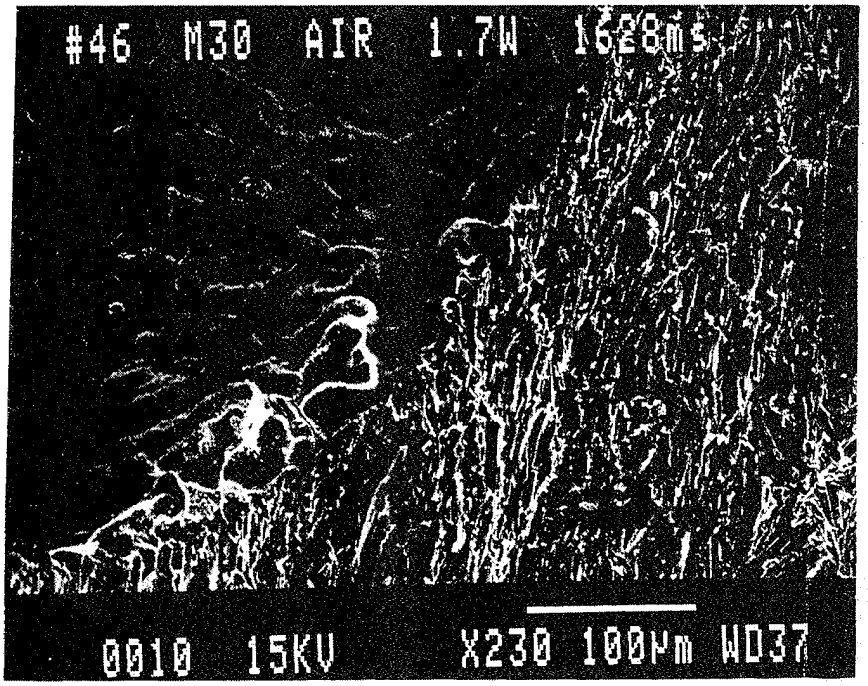

Figure 4. Cross section of melt layer of laser-irradiated grain of M30 propellant.

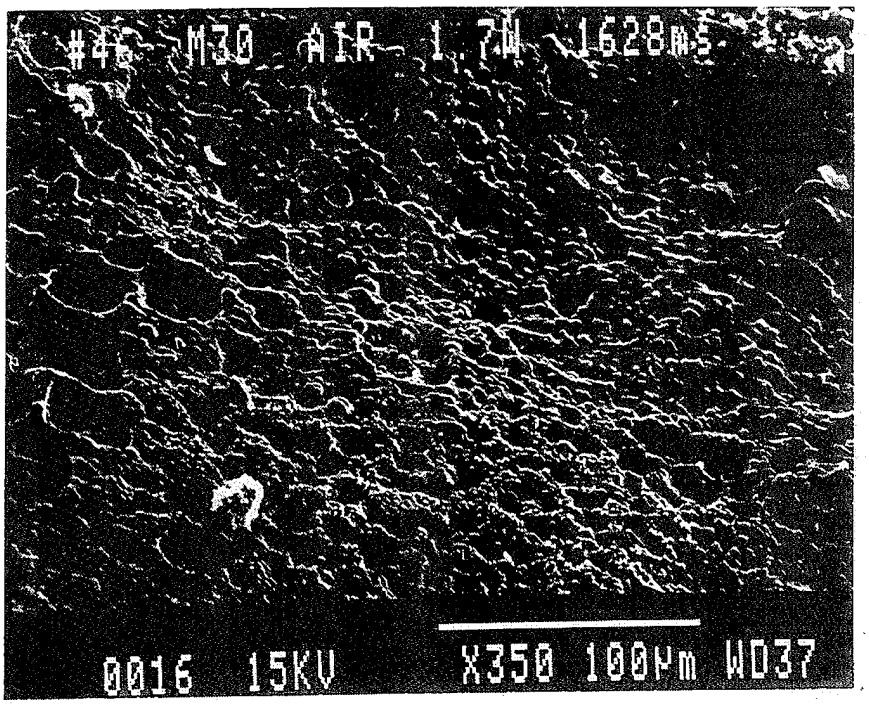

Figure 5. Top of melt layer of laser-irradiated grain of M30 propellant.

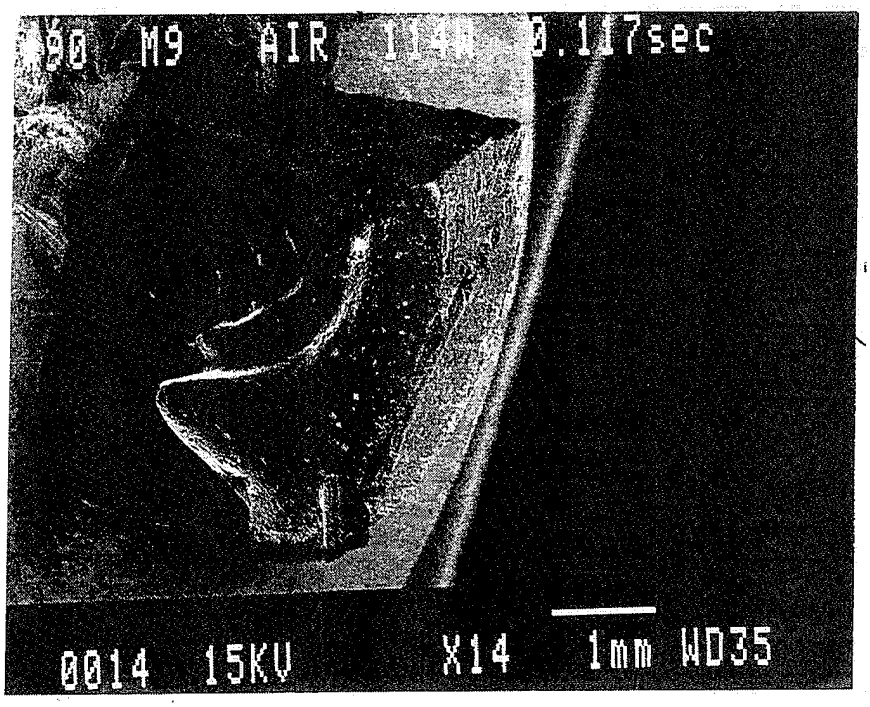

Figure 6. Overall view of portion of laser-irradiated grain of M9 propellant.

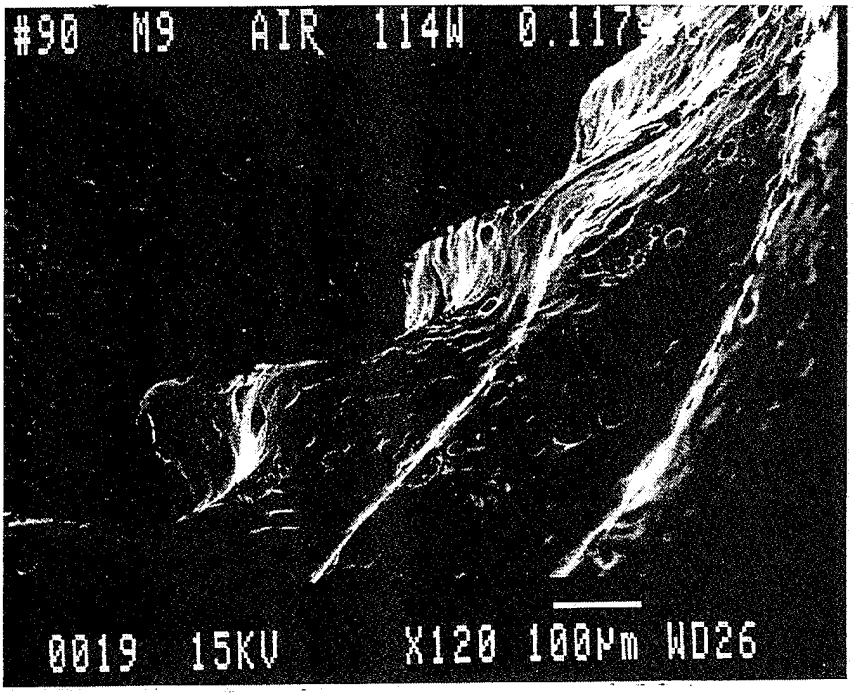

Figure 7. Cross section of surface layers of laser-irradiated grain of M9 propellant.

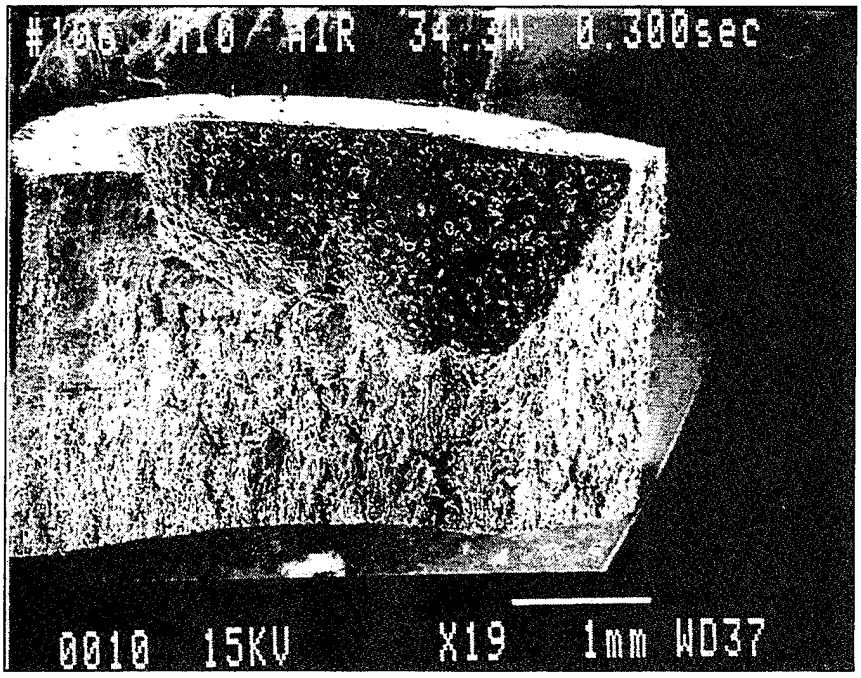

Figure 8. Overall view of portion of laser-irradiated grain of M10 propellant.

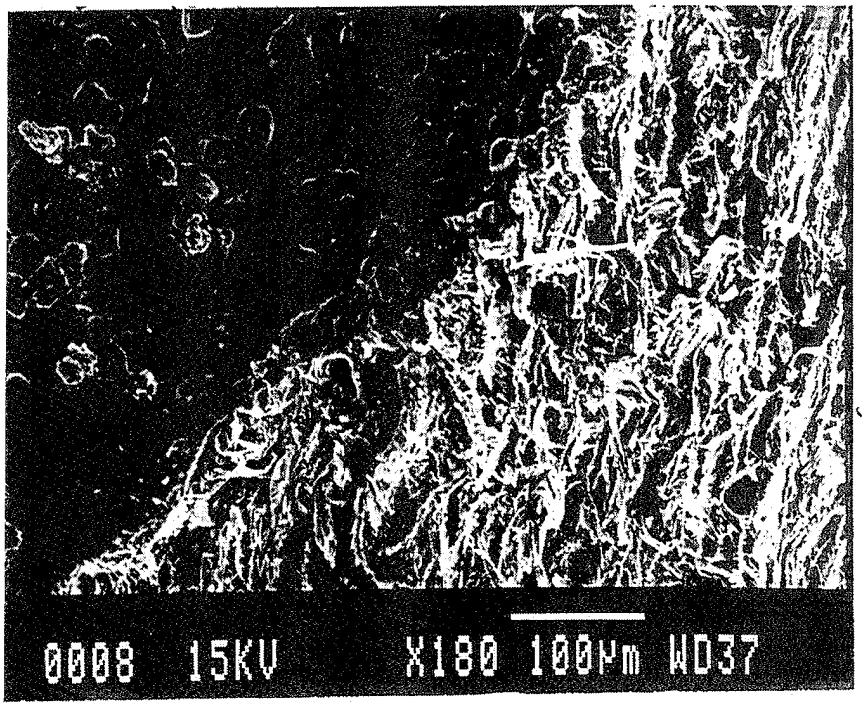

Figure 9. Cross section of surface layers of laser-irradiated grain of M10 propellant.

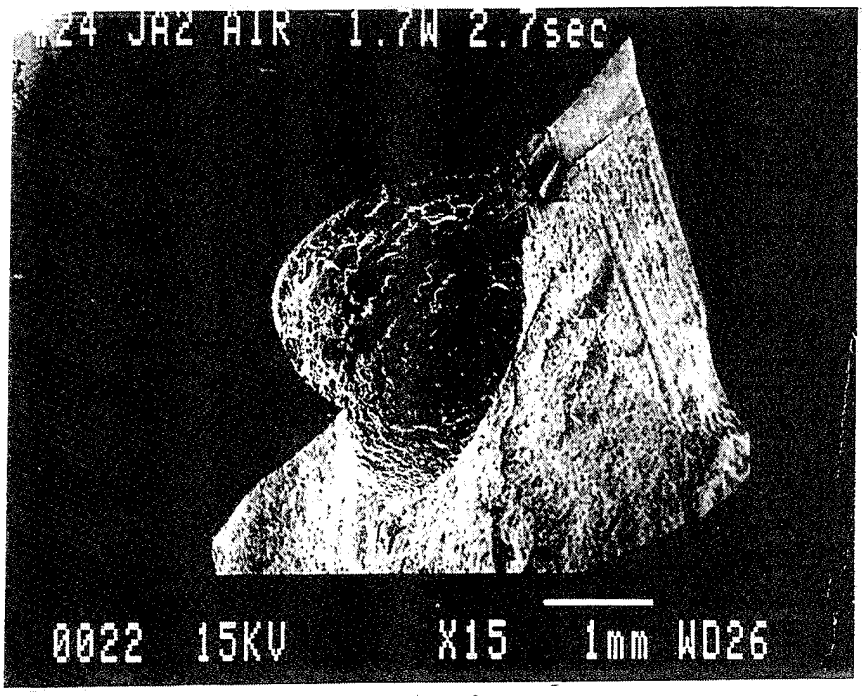

Figure 10. Overall view of portion of laser-irradiated grain of JA2 propellant.

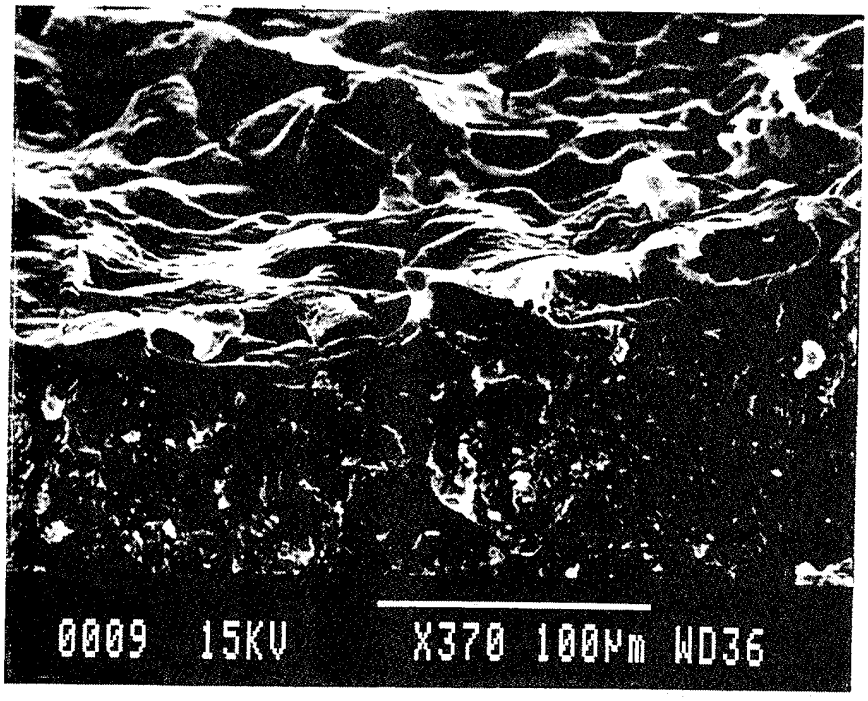

Figure 11. Cross section of surface layers of laser-irradiated grain of JA2 propellant.

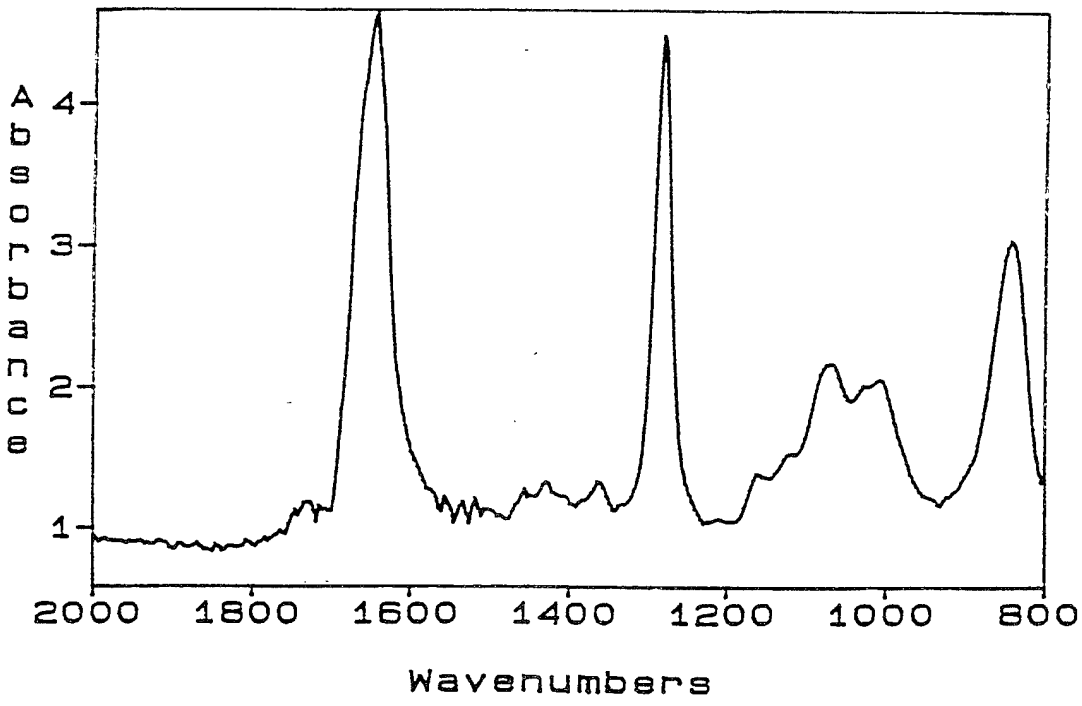

Figure 12. FTIR microscope spectrum of irradiated portion of grain of M9 propellant.

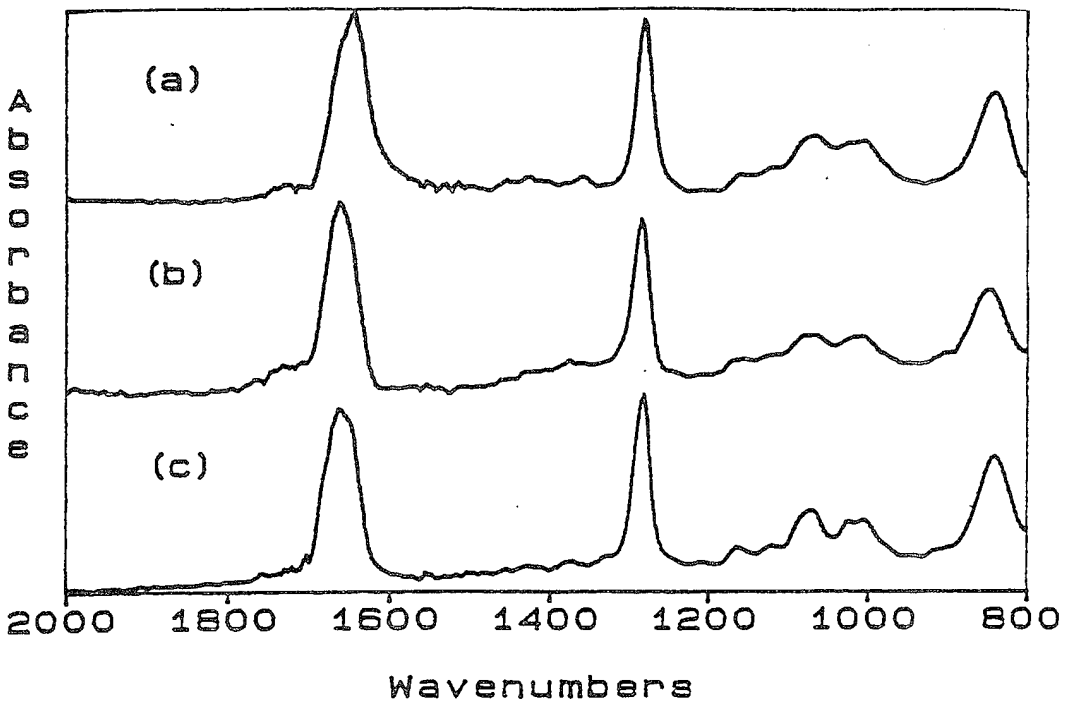

Figure 13. FTIR microscope spectra of (a) laser-irradiated M9 (sample no. 115, Table 2) propellant; (b) burned/extinguished M9 propellant (Schroeder et al. 1994); and (c) virgin M9 propellant.

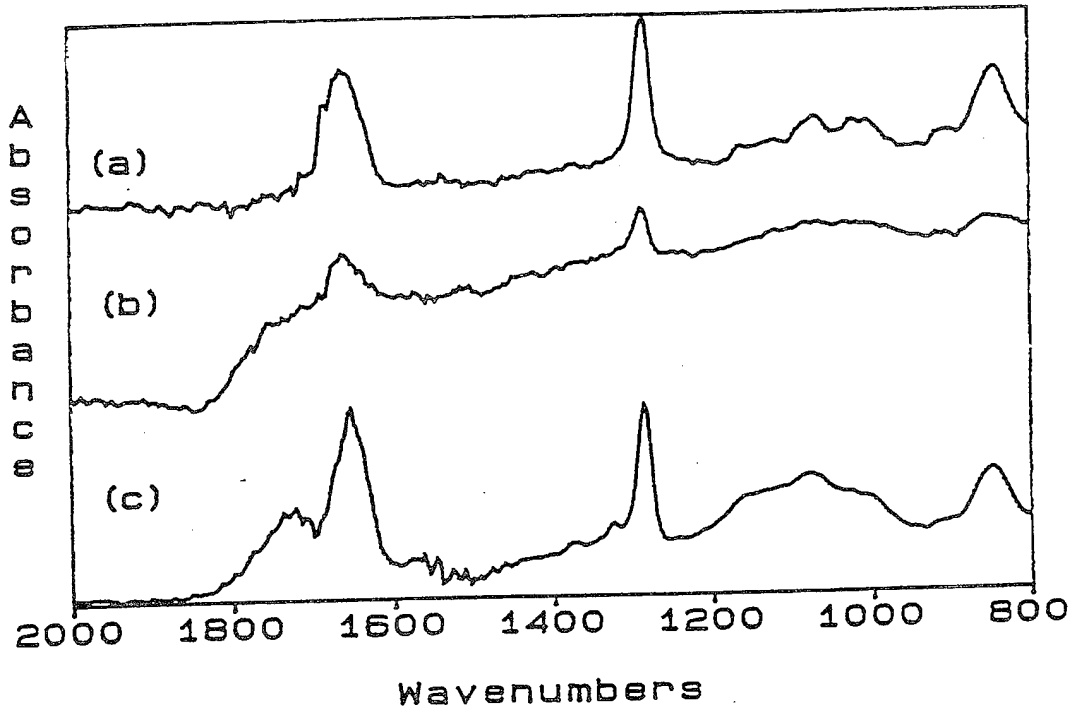

Figure 14. FTIR microscope spectra of (a) virgin JA2 propellant; (b) laser-irradiated JA2 propellant (sample no. 24, Table 2); and (c) burned/extinguished JA2 propellant (Schroeder et al. 1994).

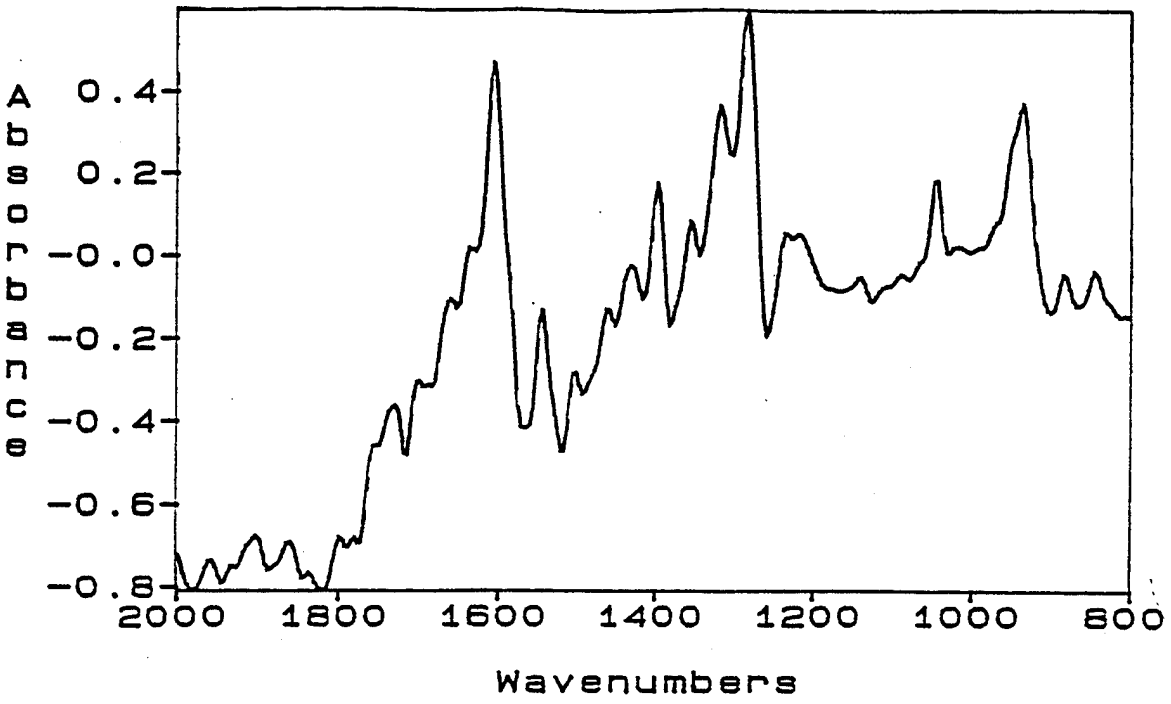

Figure 15. FTIR microscope spectrum of virgin XM39 propellant.

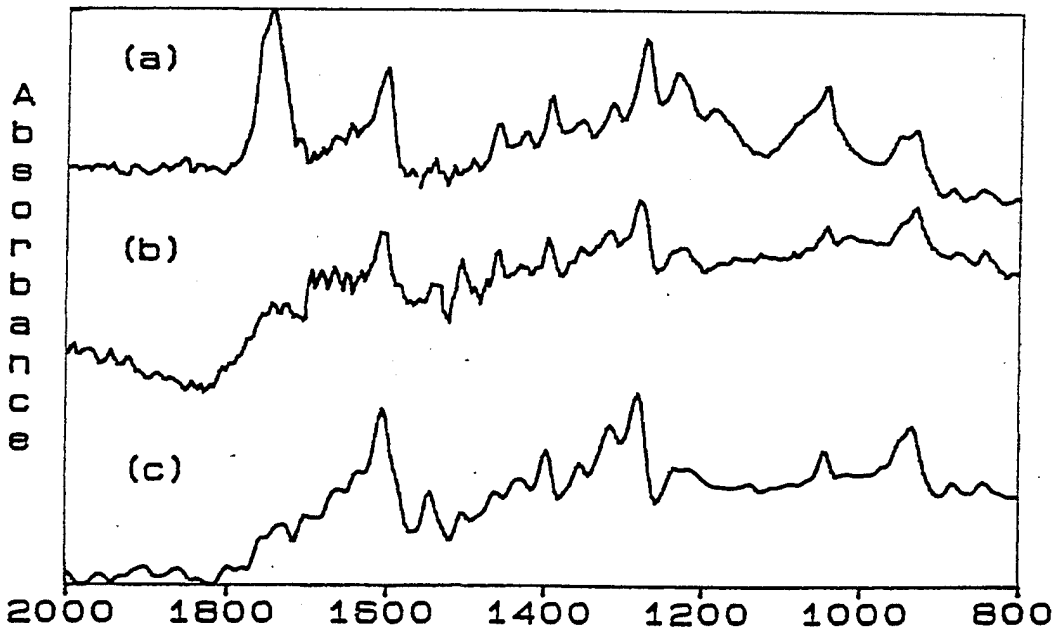

Figure 16. FTIR-microscope spectra of (a) laser-irradiated (sample no. 58, Table 2) XM39 propellant; (b) laser-irradiated (sample no. 33, Table 2) XM39 propellant; and (c) virgin XM39 propellant.

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